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WITHDRAWN FOR EXAMINATION M. L.

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H. W. WILEY, Chief.

APR 1
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METHODS FOR THE DETECTION OF RENOVATED BUTTER.

[Cooperative Work, Association of Official Agricultural Chemists, 1905.]

MELTING TEST.

Put some of the butter in a test tube or narrow beaker and heat in the boiling water oven or, to obtain the clearest possible results with rancid butter, in an oven at 60° to 70° C. Observe after one-half hour to one hour. Renovated butter always melts turbid, while fresh genuine butters that have not been melted, mixed, or ladled melt with a clear fat—with the following exceptions: Some rancid butters melt with a cloudy or sometimes even a turbid fat, and (as only recently observed) some sweet cream butters, especially unsalted ones, melt with a cloudy or even a very turbid fat, as turbid as that of a renovated butter. Perhaps any unsalted butter will behave in this way—time has not allowed of settling this point.

In making this test the tubes must not be shaken after the melting has begun; such treatment would cause any butter to melt with a cloudy or, more likely, a turbid fat.

In reporting results by this test use the term "cloudy" (little or very) in describing fats that are not perfectly clear but are still transparent, and the term "turbid" for those still further removed from the clear condition.

BOILING TEST.

a. WITH USE OF A TEST TUBE.[1]

Put into a 6-inch test tube of three-fourths inch diameter enough of the butter to make a depth of 1¼ inches when melted and boil intermittently over a full flame, boiling briskly for six or eight seconds (shaking frequently while bringing to a full boil), then remove from the flame for observation, without shaking, during an equal or greater length of time—long enough in any case to be certain of its behavior—and repeat the operation until the water is all expelled. If the butter be genuine a time arrives when, *after removal from the flame*, it boils up in small bubbles, producing a foam which rises to a greater or less height, sometimes even overflowing the tube. Very old butters, ladled butters, and, as the writer has recently noticed, unsalted butters are likely to yield only moderate or even scanty amounts of foam. In the case of unsalted butters it was found that after working into them a small amount of salt they yielded the usual abundant foam of ordinary butters. Since no such result follows from working salt into renovated butters, this fact may prove useful in rendering the result by the boiling test more distinct in certain doubtful cases. Whether the addition of salt has the same effect upon very old or ladled butters which yield scanty foam has not ye been determined by experiment, but if the butters are already well salted such a result would not be expected.

[1] U. S. Dept. of Agr., Bureau of Chemistry, Bul. No. 67, p. 115.

Renovated butters yield no foam or but very little, and boil noisily, usually with more or less of a crackling sound. Oleomargarin yields no foam and boils noisily, though often with less of the crackling sound than does renovated butter. The writer has recently tested a half dozen samples of oleomargarin, from different factories, which, although boiling noisily, with a rushing sound, made but little of the true "crackling" noise. The crackling is largely controlled by the amount of salt present—a large amount of salt reduces or prevents the crackling.

Genuine butters boil with comparatively little noise; they can usually be said to boil "quietly." Only rarely does a genuine butter emit any crackling sound, and then but little, sometimes just before the end of the boiling. This has been noticed especially with unsalted butters, but even at their worst these will not be mistaken for renovated butters.

b. USING A SPOON (TABLESPOON OR DESSERT-SPOON).

Place in a tablespoon or dessert-spoon a piece of butter the size of an ordinary chestnut, heat over a bunsen flame, at the same time stirring (with a match) until melted, then boil briskly.

Genuine butters of the ordinary salted variety boil with little or no sputtering, and yield a more or less abundant foam, persistent when the test is removed from the flame. Unsalted butters yield less foam, and frequently sputter after the manner of renovated butter; but unlike the latter, after salt has been worked into them they behave like ordinary butters.[1]

Renovated butter sputters vigorously and yields almost no persistent foam. Oleomargarin sputters similarly and yields no persistent foam.

In reporting results obtained by the boiling test state which form of the test was used—if both were used, give results separately, of course—stating whether there was "crackling" in the (a) form of the test, "sputtering" (possibly also crackling) in the (b) form, and describing the amount of foam in each—whether very abundant, abundant, moderate, scant, and so on.

MICROSCOPIC EXAMINATION.

MOUNTING.

Place the volume of one to two pinheads of butter from a freshly cut surface upon a slide and press it out quite thin under a cover glass, avoiding any lateral motion. Put three or four such mounts upon the slide; and since the particles of butter are so small, it is better to study at least six mounts, two slides, in order to get correct results.

a. LIGHT FIELD.

Observe as to smoothness or roughness; as to the abundance, size, and color of the casein spots, if any are seen, and whether they are mostly dry or inclosed in water globules. Report any other appearances observed.

Remarks.—Renovated butter, as ordinarily made, shows (like oleomargarin made similarly) numerous casein spots, large and small, appearing of a pale yellow color in the light field. "Country butters"—dairy butters by old methods—frequently show large numbers of small casein spots, while creamery butters (including, of course, dairy butter by creamery methods) show but few, if

[1] The foam from a genuine butter may often be made more abundant, especially if a rather low flame is used, by removing the boiling sample from the flame and at the same time stirring it for an instant with a match, not neglecting the outer edges. Indeed, by the simple expedient of stirring the melt. to facilitate the formation of foam, the test may be executed with only the heat of a kerosene lamp. See Household Tests for the Detection of Renovated Butter and Oleomargarine, U S. Dept. Agr., Farmers' Bulletin No. 131.

any. The writer has noticed that in many instances the casein spots of country butter are mostly dry while those of renovated butter are many of them inclosed each in a globule of brine.

b. DARK FIELD (LIGHT FROM BELOW SCREENED OFF).

Observe the casein spots, whether appearing white and gelatinous or white and glistening. The latter condition has been observed in different samples of one brand of pasteurized butter, perhaps two.[1] Whether this appearance was the result of pasteurization, or was due to other cause, is not yet known.

c. WITH POLARIZED LIGHT, NICOLS CROSSED.

Look for fat (or fatty acid) crystals, perfect or imperfect. Imperfect crystals are designated as flakes, frostiness, or haziness, according to appearance.

Remarks.—In the earlier days of the renovated butter industry the melted fat, mixed with milk, was not cooled or "granulated" as rapidly as at present, and consequently the finished product would usually show abundant evidence of having been melted, in the form of white flakes on a dark ground when examined in the manner here described. But to-day, as for some few years past, it is a rare thing in the writer's experience to find a newly made renovated butter in which these flakes can be detected, or which exhibit a greater degree of "frostiness" or "haziness" than any butter is likely to exhibit after becoming very rancid. This is, of course, because the butter fat by the present mode of manufacture is cooled too rapidly to allow it to assume crystalline form or, at least, crystalline form discernible by this mode of examination.

The frostiness and flakes which are liable to be found in any very rancid butter and which are probably free fatty acids liberated by decomposition of the glycerids, must not be mistaken for evidence that the fat has been melted. Experience will enable one to distinguish usually between the appearances due to age and those due to melting; therefore it is best to make this examination upon all butters submitted for judgment, since some renovated butters may still be thus detected, and also since foreign fat crystals can sometimes be thus detected in adulterated butters.

Similar improvement has been made in the manufacture of oleomargarin, so that to-day it is by no means uncommon to find oleomargarins which show, when examined in the way described, none of the white flakes (fragmentary fat crystals) which formerly always characterized this product. However, to completely prevent the crystallization of the fats when making oleomargarin is without doubt more difficult than when making renovated butter, for the reason that the fats of oleomargarin solidify more slowly when subjected to cold than does butter fat under the same conditions. Proof of this is seen in the behavior of the respective fats in the Waterhouse test at ice-water temperature.[2]

d. WITH POLARIZED LIGHT AND A SELENITE PLATE, BLUE AND YELLOW, BELOW THE STAGE.

Observe colors on the blue field. Purple (or violet-red perhaps) in coarse mottles or patches is nearly always seen with rancid butter of whatever kind. Fresh renovated butter may or may not show it. Therefore, this appearance signifies nothing of importance. Its presence or absence should, however, be reported to indicate the condition of the butter. Look for other colors in connection with any supposed fat crystals that may have been discovered with crossed

[1] U. S. Dept. Agr., Bureau of Chemistry, Bul. 90, p. 78. Report of referee on dairy products.
[2] U. S. Dept. Agr., Farmers' Bulletin No. 131, pp. 8, 9.

nicols alone. Red, pink, or flesh color, brass-yellow, and green may in such cases appear on the blue field.

e. EXAMINATION WHILE MELTED.[1]

Place the slide in an oven at 60° to 70° C. for five to ten minutes and examine immediately, with plain light, with and without substage illumination, i. e., in the "light field" and "dark field" as these terms are used above.

Observe the condition of the curd—whether it is in moss-like, or straggling, or fleecy forms (the latter showing only fine particles in the dark field), or circles; or whether it is in comparatively large and dense solid particles or lumps, without tendency to cohere. If only fleecy forms or light fine particles are seen, repeat the test, with mounts a little thicker than are best for the other microscopic examinations; by this means either moss-like or straggling forms are sometimes made to appear.

In genuine butter the lighter forms of curd of course appear; but why sometimes moss-like forms, sometimes straggling forms, sometimes only circles, fine particles and fleecy forms, the writer is unable to say. All that can be said is that observations to date indicate pasteurization (of the cream) as one of the causes tending to destroy the cohesive power of the curd of genuine butter, and thus to prevent the moss-like grouping which commonly occurs in unpasteurized creamery butters. Other causes there certainly are, among which it seems not improbable that the degree of ripening of the cream may be one, and possibly also the amount of salt present, but upon these points no special observations have been made.

In renovated butter, on the other hand, as it is ordinarily made, a large part of the curd is in solid lumps or particles of considerable size without tendency to cohere, that is, in the form of "coarse curd."

Something resembling this appearance, though usually much less pronounced, is sometimes seen in badly made "country butters" from which the butter-milk has not been properly washed in making; but there is seldom any danger of mistaking the one kind of butter for the other.

The only real danger of being misled by this test (e) is when one has to deal with a renovated butter made without the admixture of milk to the butter fat, in which case, naturally, "coarse curd" is not likely to be present. The writer has met with but two samples of such butter, both from the same factory. They were fully proved to be renovated butters by the other tests (not microscopic), but "coarse curd" was entirely absent, as well as "casein spots" in the light and dark field examinations, (a) and (b) above. As protein matter was present in about normal quantity, it is believed that this butter was made by churning the purified butter fat with *cream* (perhaps with previous emulsification of the butter fat therewith) and subsequently washing, as in regular butter making, thus causing the microscopic appearance to somewhat resemble that of genuine butter. Indeed, the microscopic appearance favored the idea that *pasteurized* cream had been used. But, however the butter was actually made, there was no doubt that it was renovated, so plainly did it show its character in the melting, boiling, and Waterhouse tests.

In reporting results obtained by the microscopic examination of the butters sent out, please report regarding all the appearances above mentioned, under a, b, c, d, and e.

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[1]This mode of observation (e) gives all the information that is furnished by the Hess and Doolittle "curd test," with much less work, and is now habitually used by the writer in place of it.

THE WATERHOUSE TEST, MODIFIED.[1]

One hundred cubic centimeters of distilled water in a small tin cup, 2¾ inches in diameter, are heated to just below 50° C., a slightly rounded teaspoonful (about 8 grams) of the butter is added, and gently stirred[2] until melted. The temperature is then raised to exactly 50°, the cup is immediately placed in the pan 9 to 9½ inches in diameter at the base, containing 1,000 cc of water at 12° C., the pan itself having been previously cooled by water to the same temperature; so that, as far as possible, the water in the pan shall have its temperature raised *only* by the water and fat in the cup. The contents of the cup are then stirred rapidly with a slender wooden stirring rod—more slender than a match and about 5 or 6 inches long—the stirring being done by both circular and crosswise motions, a number of the former alternating with a larger number of the latter, in such manner as to keep the *whole mass in lively motion, and at the same time to prevent if possible the formation of a coherent mass of fat at the center.* A little experience will show exactly how this is done. The stirring stick should be held somewhat inclined, by keeping the hand to one side of the cup instead of directly over it, in order to avoid rubbing off from the sides of the cup any fat that may adhere. The water in the pan is well stirred at least once every minute, by moving the cup about. The stirring of the contents of the cup need never be continued longer than ten minutes, and frequently a definite result is obtained in a shorter time.

If the butter granulates it is *not* renovated. Renovated butter gathers, after three to five minutes' stirring, into a "soft flat mass," which, at first small, grows with continued stirring until it nearly or entirely covers the surface of the water in the cup when the stirring is stopped for a moment or two. This stage is reached at about six minutes from the start. The stirring being continued, the "flat mass" changes into a very "soft lump," leaving the water nearly or quite clear, in seven to ten minutes from the start. These, at least, are the times that have been observed on samples tested since these details of the test have been carefully observed; other samples may possibly change the figures slightly.

But, as stated in former reports, one occasionally finds an entirely genuine butter that gathers in the test (carried out under the temperature conditions specified), such butters having usually been found among either dairy butters, made from gravity cream, or pasteurized butters. And from these two kinds of butter, portions from the same lot have given different results in the hands of different experimenters and of the same experimenter at different times. This was the case with the two dairy butters and the two pasteurized butters used in the association work of 1904. For these contradictory results a possible explanation was suggested in the theory that the change undergone by the samples in a few weeks' keeping might have changed their behavior in the test. While this possibly may be the true explanation, it now seems probable that a detail of manipulation in the preliminary part of the test, heretofore not much considered, may prove the chief factor in producing these discordant results. In the directions for carrying out the test it has always been stated that after the spoonful of butter has been added to the 100 cc of water at 50° C. it is to be "gently stirred until melted." Now whether the difference between "gentle" stirring and "rapid" stirring at this point would materially affect results the

[1] The directions here given are the same as those in the writer's reports to the association (Bul. No. 67, p. 127, and Bul. No. 81, p. 81, U. S. Dept. of Agr., Bureau of Chemistry) with the addition of certain details of procedure and observation which experience has indicated.

[2] See suggestion as to *rapid* stirring, page 6.

writer can not say, but he has recently observed that the entire *omission* of stirring during the time of melting will, in some cases at least, cause even a genuine creamery butter to "gather in a soft lump" after eight or ten minutes of the subsequent cooling and stirring, the appearance before stirring being very oily—in short, will cause it to behave considerably like a renovated butter. This is not surprising, for the slow, quiet melting allows a more or less complete separation of the oil from the curd.

It was found, on the other hand, that by submitting a renovated butter to *very rapid* stirring during the time of melting, while the main result was the same as usual with renovated butter, the melted mass was very much less oily in appearance than usual—that is, less oily than when stirred *gently*, or when left with almost no stirring during the melting, as no doubt was sometimes done in routine work before special attention was given to this detail.

From these facts it seems quite probable that some of the conflicting results heretofore obtained on the same butter, and the different appearances of the charge as regards oiliness when melted on the surface of the water, may be due to differences in the rapidity or the persistence of stirring during the time of melting. The writer has had no time to investigate this point, and he therefore suggests that the workers this year make observations upon the same and report results. At the present time it seems reasonable to expect the most useful results from *rapid* stirring during the melting, since in the few trials made by the writer that treatment did not change the main result with renovated butter, and since *very* gentle stirring would probably, in some cases at least, entirely change the main result with genuine butter, just as entire omission of stirring did with two out of three of those experimented with by the writer. Therefore, it is suggested that *very rapid* stirring during the time of melting be given a trial in this year's work, in comparison with the gentle stirring prescribed in the directions.

In reporting upon the butters sent out, the following points should be covered:

(1) General result, i. e., whether the butter granulated or gathered.

(2) If it gathered, the number of minutes after stirring commenced that it could be surely said to have begun gathering in a "soft flat mass," that is, when the mass is about the size of a silver quarter-dollar.

(3) The number of minutes from beginning of stirring until the "soft flat mass" assumes the form of a "soft lump;" and the temperature of the water in the pan and in the cup at that moment.

(4) Whether or not the butter presented an oily appearance when melted upon the water at the beginning of the test or during the first minute or so of the cooling and stirring.

Results of all the tests, with remarks or suggestions regarding the same, should be sent to the referee at as early a date as possible.

G. E. Patrick,
Referee on Dairy Products,
Chief, Dairy Laboratory.

Approved:
James Wilson,
Secretary of Agriculture.

Washington, D. C., *October 9, 1905.*

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